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UNIVERSITY OF WATERLOO
WATERLOO RESEARCH INSTITUTE
WATERLOO, ONTARIO

CONTRACT REPORT NO.OSQ82-00187

RADIOCHEMICAL DETERMINATION OF TAILINGS REFERENCE MATERIAL

WRI PROJECT # 210-08

DSS FILE NO. 15SQ.23440-2-9145-6

D R A F T
F I N A L R E P O R T

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MICROMEDIA

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1.1 The tailings samples each weighing approximately 2 grams were heated with potassium pyrosulfate to a clear melt after each sample had been treated with hydrofluoric acid and sulfuric acid for the removal of SiO_2. The melt was dissolved in 10 percent hydrochloric acid. The solution was made to 100 ml.

1.2 Analytical Separations. A procedure for the simultaneous separation of thorium, radium and lead as outlined in our laboratory by E. Veska, was followed. To an aliquot of the solution ^{234}Th was added as a tracer for evaluating chemical recovery of thorium. The solution was adjusted to pH of 2-3 and then passed through a cation-exchange resin column, at a flow rate of 1.5 ml per minute. All radionuclides of interest were retained in the column. The exchange capacity of the resin was kept sufficiently high so as to retain all cations in the column. 200 ml of 12M HCl was passed through the column which removed all radionuclides of interest with the exception of thorium. Thorium was eluted subsequently with 200 ml of 0.5 M oxalic acid.

The preparation of purified thorium source was achieved by oxidizing oxalate ions with nitric and perchloric acid completely and subsequent extraction of thorium 0.25M thenoyltrifluoroacetone in benzene from the solution adjusted to pH of .5 and back extraction to aqueous phase with 2M nitric acid. Thorium was electrodeposited on a planchet according to the technique described by Puphal and Olson. The alpha-particle spectrum from the sample was recorded by using a solid-state detector and a pulse-height analyzer. Three peaks due to alpha particles from ^{228}Th, ^{230}Th and ^{232}Th could be identified easily and the number of events under each peak provided data for the evaluation of the activity due to each isotope. The efficiency of the detector and the geometry factors were determined by a calibrated source of ^{241}Am obtained from the National Bureau of Standards, Washington, D.C. USA.

The eluant containing radium, bismuth lead and other cations was boiled and made to IM in HCl. 1 ml of xylene was added to the solution prior to storage for a period of a week. Lead and bismuth were separated by extraction with diethylammonium diethyldithiocarbanate from radium. The organic phase was washed with 1 M HCl and then deposited on a planchet for counting. The sample was counted twice by using a low background proportional counter 20 minutes and 1 day after the extraction. The counter was calibrated by using standard ^{226}Ra and ^{210}Pb sources. The activity due to the ingrowth of daughters of ^{226}Ra was then used for the evaluation of activity of ^{226}Ra by following the standard decay and growth equations.

From an aliquot of the solution of the melt, radium was coprecipitated with lead and barium as sulfate. ^{133}Ba was added to monitor the chemical recovery of radium from the solution. The precipitate was dissolved in diethylenetriamine pentaacetic acid and barium sulfate reprecipitated by the addition of 6M acetic acid. The precipitate was redissolved and allowed to stand for a minimum of three days. ^{228}Ac was separated by using TTA and its activity assayed by the low background proportional counter. The activity of the parent ^{228}Ra was evaluated by using the standard decay and growth equations. The activity of ^{228}Ra is still to be determined for standard samples. The data have been acquired and the activity of this isotope will be reported as soon as the detector has been standardized for the detection of beta particles from ^{228}Ac.

For the separation of polonium, the solution was adjusted to pH = 0.3 and then placed in sample holder fitted with a silver foil. The solution was heated to 95°C and stirred for several hours to allow the deposition of polonium on the foil. The foil was assayed for its activity by using the alpha spectrometer.

The results are presented in the attached table. However, these results may be regarded as preliminary ones. The detectors for radiochemical assay and the chemical procedures for elemental separations are being examined and final results will be reported before the end of April.

SAMPLE	^{210}Po pCi/g	^{226}Ra pCi/g *	^{210}Pb pCi/g *	^{230}Th pCi/g	^{232}Th pCi/g	^{228}Th pCi/g	^{228}Ra pCi/g	Subsample Size Masses(g)
	52.6 ± 2%	46.6 ± 2%	49.6 ± 2.3%	49.4 ± 1.7%	9.09 ± 1.9%	9.61 ± 2.1%	BEING	2.00155g
UTS-1	47.1 ± 3%	47.87 ± 2%	49.3 ± 2.0%	54.9 ± 1.6%	10.54 ± 2.0%	12.0 ± 2.0%	EVALUATED	2.0000
(April 1)	49.8 ± 2%	43.4 ± 2.1%	52.8 ± 2.0%	51.2 ± 1.7%	9.87 ± 2.0%	10.65 ± 2.1%	AS STATED	2.00175
	52.9 ± 2%	46.2 ± 2%	49.0 ± 1.8%	52.3 ± 1.7%	9.95 ± 2.0%	10.9 ± 2.0%	IN THE	1.99870
	103.0 ± 1.8%	65.4 ± 1.8%	57.8 ± 2.0%	248.0 ± 1.8%	45.9 ± 2%	36.2 ± 1.9%	REPORT	2.00085
UTS-2	94.2 ± 2%	69.5 ± 1.8%	≠	200.0 ± 1.8%	46.3 ± 2%	34.3 ± 1.9%		2.00125
(April 2)	106.2 ± 2%	70.0 ± 1.9%		257.2 ± 1.7%	48.9 ± 2%	34.8 ± 1.9%		2.00065
	100.5 ± 1.9%	62.5 ± 2%		252.6 ± 1.8%	48.2 ± 2%	35.1 ± 2.0%		1.99950
	171.7 ± 1.9%	426.6 ± 2%	157.6 ± 1.8%	172.2 ± 1.3%	---	---	---	2.00015
UTS-3	164.7 ± 2.2%	396.3 ± 2%	159.4 ± 1.8%	183.5 ± 1.4%	---	---	---	2.00095
(April 3)	172.7 ± 1.8%	392.2 ± 1.9%	165.2 ± 1.7%	175.4 ± 1.4%	---	---	---	2.00065
	161.9 ± 2%	410.2 ± 2%	153.0 ± 2%	177.3 ± 1.3%	---	---	---	1.99970
	380.0 ± 1.2%	459.2 ± 1.9%	321.8 ± 1.6%	342.7 ± 1.0%	---	---	---	2.0000
UTS-4	342.0 ± 2%	452.7 ± 1.8%	316.9 ± 1.6%	325.3 ± 1.5%	---	---	---	1.99970
(April 4)	362.2 ± 2%	419.5 ± 1.8%	300.8 ± 1.7%	315.5 ± 1.6%	---	---	---	2.00030
	369.5 ± 1.9%	411.2 ± 1.5%	311.0 ± 1.8%	320.4 ± 1.5%	---	---	---	1.99985
	168.2 ± 2.1%	120.6 ± 1.9%	≠	220.1 ± 1.9%	26.4 ± 1.9%	28.5 ± 2%		2.00015
UTS-5	154.6 ± 1.8%	108.9 ± 2%		228.7 ± 1.8%	29.5 ± 1.8%	26.6 ± 1.9%		2.00095
(April 4)	143.8 ± 2.3%	128.7 ± 1.9%		219.2 ± 1.8%	29.8 ± 1.8%	26.8 ± 1.9%		2.00065
	142.0 ± 2%	123.6 ± 1.9%		225.6 ± 1.9%	30.4 ± 1.9%	27.3 ± 1.9%		1.99970

* RESULTS ARE BEING CHECKED

≠ RESULTS ARE BEING PROCESSED

5-43.1b

EMAR OTT

U OF W WTLO

JUNE 6/83

ATTN: DR CLINT SMITH
EMR

PLEASE REPLACE THE TABLE IN OUR EARLIER REPORT WITH THE FOLLOWING AND CONSIDER IT AS OUR FINAL REPORT ON WRI PROJECT NO 210-08 DSS FILE NO 15SQ.23440-2-9145-6.

NUMBERS IN BRACKET REPRESENT PER CENT ERROR. THE ERROR IS TAKEN AS ONE SIGMA.

	210	226	210	230
	PG	RA	PB	TH
	BO/G	BO/G	BO/G	BO/G
UTS-1	3.48(2.2)	3.79(2.1)	3.43(2.6)	3.50(2.0)
	3.44(2.5)	4.04(1.5)	2.52(3.0)	3.63(2.2)
	3.67(2.9)	3.59(2.2)	3.73(2.5)	3.77(2.2)
	3.66(2.9)	4.15(2.2)	3.09(2.7)	3.79(2.1)
UTS-2	4.11(1.9)	5.35(1.8)	3.54(2.0)	6.22(2.1)
	3.85(1.9)	4.72(1.7)	3.47(1.9)	6.66(2.0)
	3.48(2.8)	5.06(1.3)	4.40(1.9)	6.77(2.2)
	3.50(2.2)	4.88(0.7)	4.85(1.8)	6.70(1.9)
UTS-3	12.8(0.9)	12.15(1.7)	12.9(2.1)	11.0(2.0)
	13.1(2.1)	11.97(2.0)	12.2(2.0)	11.3(1.7)
	12.3(2.2)	12.90	12.1(2.0)	10.2(1.7)
	12.3(2.0)	13.40	11.0(2.1)	10.4(0.9)

UTS-4	27.9(2.1)	40.5	26.7(1.9)	23.6(2.0)
	27.4(1.5)	39.4	26.1(1.9)	26.2(1.7)
	26.9(1.1)	42.7	24.9(2.0)	24.1(1.6)
	27.6(1.9)	40.7	27.5(1.8)	24.7(2.0)
UTS-5	11.9(0.9)	16.1	10.6(2.0)	13.0(2.0)
	12.4(1.8)	15.3	9.7(2.1)	12.4(1.8)
	12.6(2.4)	15.1	8.5(2.0)	13.4(2.0)
	10.1(2.8)	15.7	11.0(1.9)	12.2(2.0)

	232 TH BQ/G	228 TH BQ/G	228 RA BQ/G	SUBSAMPLE G
UTS-1	0.75(2.1)	1.22(2.2)	1.0(3.2)	
	0.70(2.0)	0.78(2.1)	0.71(2.9)	2.00 G
	0.73(2.0)	0.77(2.2)	0.82(2.8)	
	0.68(2.2)	0.66(2.3)	0.79(2.7)	
UTS-2	0.91(3.0)	1.23(2.1)	1.11(2.9)	
	0.91(3.5)	1.29(3.0)	1.21(3.0)	2.00 G
	1.27(3.1)	1.21(3.0)	1.17(2.9)	
	1.26(2.7)	1.44(2.6)	1.09(2.8)	
UTS-3				2.00 G
UTS-4				2.00 G
UTS-5	1.82(3.1)	1.61(3.0)	1.75(2.8)	
	1.46(3.0)	1.53(3.0)	1.81(2.9)	2.00 G
	2.01(3.2)	1.84(3.1)	1.72(2.6)	
	1.35(3.1)	1.87(3.0)	1.81(2.7)	

ADD MISSING NUMBERS IN BRACKETS FOR RA-226 AS FOLLOWS FOR UTS-3, UTSS-4 AND UTS-5:

UTS-3 1.7, 0.8

UTS-4 0.6, 0.8, 0.4, 0.8

UTS-5 1.3, 1,4, 1.4, 1.3

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